



United States Department of the Interior

U.S. GEOLOGICAL SURVEY
National Water Quality Laboratory
P.O. Box 25585
Denver, CO 80225-0585

NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 2015.01

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Subject: Modification to the reporting of elemental results for whole-water recoverable (WWR) sample analyses

Effective: May 1, 2015

Author(s): Sarah J. Stetson, Methods Research and Development, 303-236-3277, sstetson@usgs.gov and John R. Garbarino, Methods Research and Development, Retired

Revision: N/A

PURPOSE

This technical memorandum describes the implementation of a correction to account for dilution during digestion of whole-water recoverable (unfiltered, WWR) samples analyzed at the National Water Quality Laboratory (NWQL), using inductively coupled plasma-optical emission spectrometry (ICP-OES) and inductively coupled plasma-mass spectrometry (ICP-MS). It also describes the procedure used to apply the dilution factor to elemental results.

BACKGROUND

The in-bottle digestion (INBD) procedure (laboratory code 1735; Hoffman and others, 1996) replaced the hot-plate digestion procedure (Fishman and Friedman, 1989) at the NWQL in April 1992 to reduce the opportunity for trace-element contamination during the acid digestion of WWR samples. In the INBD procedure, an aliquot of concentrated hydrochloric acid equal to 2 percent of the weight of the sample is added to the sample. The acidified sample is then digested in an oven at 65 °C for 8 hours.

The NWQL has determined that the accuracy of elemental results for WWR samples analyzed by ICP-OES and ICP-MS can be improved by accounting for the sample dilution associated with the INBD. Historically, the sample dilution resulting from the addition of the acid has not been

taken into account when reporting results. The error associated with this exclusion was thought to be insignificant, considering the unknown contribution of the sediment mass present in the sample as well as other practices in the INBD procedure that contribute to the propagation of error. The 2 percent negative bias may be important, however, when comparing filtered and unfiltered sample results. This is especially critical as elemental concentrations increase and considering that approximately 80 percent of whole-water samples submitted to the laboratory contains less than 100 milligrams per liter (mg/L) of sediment (Rus and others, 2012).

SCOPE

Beginning May 1, 2015, all elemental results from WWR by ICP–OES and ICP–MS analyses will be adjusted prior to reporting to account for the 2 percent sample dilution resulting from the addition of the acid. This change will affect all analyses performed on or after May 1, 2015.

To adjust for the dilution introduced during the digestion of WWR samples, calibrator nominal concentrations will be adjusted up by 2 percent prior to quantitating WWR sample data. For example, for a calibrator made to 1.0 mg/L, 1.02 mg/L would be used as the concentration in the calibration calculations. This will be accomplished by multiplying calibrator concentrations by 1.02 and using the adjusted concentrations to calculate sample concentrations.

Elemental results acquired prior to May 1, 2015 will not be corrected and reloaded. The magnitude of the data correction is considered small enough not to outweigh the time and effort associated with a data reload for all data associated with the introduction of the INBD process. This is a large amount of data that has the potential to affect many projects. A reload may also lead to confusion among data users. A reload of this magnitude would create a cascade effect since all data that have been used in numerous project reports and other publications would also have to be corrected.

There will be approximately a 2 percent shift in the data for laboratory codes listed in table 1 reported on or after May 1, 2015. Data users will need to consider this shift in data when comparing data from before and after the implementation of the 2 percent correction and identify it in interpretive reports.

Table 1. Laboratory codes affected by the modification in reporting elemental concentrations in whole-water recoverable (unfiltered) sample results from inductively coupled plasma–optical emission spectrometry (ICP–OES) and inductively coupled plasma–mass spectrometry (ICP–MS).

[ICP–OES, inductively coupled plasma–optical emission spectrometry; ICP–MS, inductively coupled plasma–mass spectrometry; not applicable, N/A]

Element	Laboratory code	
	ICP–OES	ICP–MS
Aluminum	2351	2372
Antimony	N/A	2373
Arsenic	N/A	3123
Barium	2352	2374
Beryllium	2353	2375
Boron	2354	2501
Cadmium	2355	2376
Calcium	2356	N/A
Chromium	2357	3127
Cobalt	2391	3125
Copper	2358	3129
Iron	2359	N/A
Lead	N/A	2380
Lithium	2361	2381
Magnesium	2362	N/A
Manganese	2363	2382
Molybdenum	2364	2383
Nickel	2365	3131
Potassium	2775	N/A
Selenium	N/A	3133
Silicon (as silica)	2366	N/A
Silver	2367	2386
Sodium	2368	N/A
Strontium	2369	2387
Thallium	N/A	2388
Titanium, custom	N/A	3083
Tungsten	N/A	3137
Uranium	N/A	2389
Vanadium	2370	3135
Zinc	2371	3139

REFERENCES

Fishman, M.J. and Friedman, L.C., eds., 1989, Extraction procedure, water-suspended sediment, Method I-3485-85, *in* Techniques of Water Resources Investigations of the United States Geological Survey, Methods for the Determination of Inorganic Substances in Water and Fluvial Sediments, Book 5, Chapter A1, p. 50.

Hoffman, G.L., Fishman, M.J., and Garbarino, J.R., 1996, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—In-bottle acid digestion of whole-water samples, U.S. Geological Survey Open-File Report 96-225, 28 p.

Rus, D.L., Patton, C.J., Mueller, D.K., and Crawford, C.G., 2012, Assessing total nitrogen in surface-water samples—Precision and bias of analytical and computational methods: U.S. Geological Survey Scientific Investigations Report 2012-5281, 38 p.

EFFECT ON DATABASE

Effect on USGS National Water Information System (NWIS) database

1. For samples analyzed on or after May 1, 2015, all data reported to NWIS will have been adjusted to account for dilution during digestion procedure.
2. No change to historical data in NWIS.



/signed/

David W. Reppert, Acting Chief
National Water Quality Laboratory
Branch of Analytical Services

Supersedes: N/A

Key words: ICP-MS, inductively coupled plasma-mass spectrometry, ICP-OES, inductively couple plasma-optical emission spectrometry, in-bottle digestion, INBD, whole water recoverable, WWR

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